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Modeling of cryogenic frictional behaviour of titanium alloys using Response Surface Methodology approach

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ABSTRACT

The potential of cryogenic effect on frictional behaviour of newly developed titanium alloy Ti–5Al–4V–0.6Mo–0.4Fe (Ti54) sliding against tungsten carbide was investigated and compared with conventional titanium alloy Ti6Al4V (Ti64). In this study, four models were developed to describe the interrelationship between the friction coefficient (response) and independent variables such as speed, load, and sliding distance (time). These variables were investigated using the design of experiments and utilization of the response surface methodology (RSM). By using this method, it was possible to study the effect of main and mixed (interaction) independent variables on the friction coefficient (COF) of both titanium alloys.

Under cryogenic condition, the friction coefficient of both Ti64 and Ti54 behaved differently, i.e. an increase in the case of Ti64 and decrease in the case of Ti54. For Ti64, at higher levels of load and speed, sliding in cryogenic conditions produces relatively higher friction coefficients compared to those obtained in dry air conditions. On contrary, introduction of cryogenic fluid reduces the friction coefficients of Ti54 at all tested conditions of load, speed, and time.

The established models demonstrated that the mixed effect of load/speed, time/speed, and load/time consistently decrease the COF of Ti54. However this was not the case for Ti64 whereas the COF increased up to 20% when the Ti64 was tested at higher levels of load and sliding time. Furthermore, the models indicated that interaction of loads and speeds was more effective for both Ti-alloy and have the most substantial influence on the friction. In addition, COF for both alloys behaved linearly with the speed but non-linearly with the load.

1. Introduction

Titanium and its alloys are characterized by lightweight, high strength, corrosion resistance and biocompatibility materials. They are widely used in many applications, such as aeronautical, military [1,2], biomaterial (for joint replacement and dental appliances) [3–5], sport equipments, defence, chemical, petrochemical and marine structure [6]. From machining point of view, pure titanium and its alloy Ti6Al4V are regarded as one of the most difficult material to machine because of their low thermal conductivity (about 6.7 W/mK) which is significantly lower than steels and their alloys (about 51.9 W/mK) [7]. The generated heats dissipate slower and cause the temperatures in the cutting tool and work piece to rise dramatically when machining these materials. As a consequent, the high temperature shortens the cutting tool life and also produces poor quality of machined surface [1,2].

In addition to machining difficulty, titanium alloys have poor tribological properties [6,8] which are attributed to their poor resistance to plastic shearing and low work hardening. These in turn influence the mechanical and the wear characteristics [8]. Besides, surface oxide (formed during dry sliding due to high flash temperatures) is easily removed by spalling or micro-fragmentation and does not protect the subsurface layers against wear. Moreover, the dissolved atmospheric oxygen tends to embrittle the matrix and thus reduces the mechanical resistance of the material [8]. Furthermore titanium has low percentage of d-bond character (27%) which means that the lesser the percentage of d-bond character, the more active is the metal and the greater the friction [6].

The poor tribological behaviour of titanium alloy is characterized by high coefficients of friction, severe adhesive wear with a strong tendency to seizing and low abrasion resistance [9]. For this reason, different approaches by different researchers have been proposed for improving tribological properties of titanium alloys. These include surface treatment [6,10,11], ion implanted [12–14], coating [15], oxygen diffusion [16] and new alloys with different composition [17]. Tribological concerns for Ti alloy in aerospace components have focused mainly on their fretting behaviour, leading to research on surface treatments like ion implantation and solid film lubrication [14]. It was reported that the best friction and

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wear results for Ti6Al4V alloys were obtained for anodized counter-surfaces coated with MoS_2 solid-film or with polytetrafluoroethylene (PTFE), but the abrasion resistance was poor.

A number of different surface modifications (treatments and coatings) techniques, such as physical vapor deposition (PVD) [2], plasma immersion ion implantation [18], thermal oxidation [4,6], plasma and laser nitriding [19], were recently applied to titanium alloys and mainly to the more widely used Ti6Al4V, in order to improve their tribological behaviour.

The influences of heat treatment, surface modification and counterface materials on the wear characteristics of new β type titanium (Ti–Nb–Ta–Zr) alloys and conventional titanium alloy Ti6Al4V sliding against stainless steel disc in a 0.9% NaCl solution were investigated in [4]. In this study, the authors concluded that the wear resistance of Ti–29Nb–13Ta–4.6Zr and Ti6Al4V is insensitive to their mechanical properties, such as hardness and ultimate tensile strength. Thus, the heat treatment approach (which improves the mechanical properties of Ti alloys) was not a practical way to improve the wear resistance of these titanium alloys.

In more recent work [20], attention was focused on controlling the heat-transfer capability of Ti alloy as a factor that affects the tribological properties of Ti6Al4V alloys. In this work, the friction and wear behaviour of Ti6Al4V alloys dry sliding against GCr15 steel were investigated on a high-speed pin-on-disc tribometer. It was concluded that enhancement of heat-transfer capability is of great benefit in reducing the sliding interface temperature for Ti6Al4V alloy and reducing the friction coefficient of Ti6Al4V/GCr15 pairs with increasing the sliding velocity.

Among the different types of titanium alloys, $\alpha+\beta$ alloys exhibit the best wear results. The better wear resistance of the $\alpha+\beta$ alloys was reported to be mainly due to the increased resistance to plastic deformation that is attributable to the existence of α needles in the retained β matrix [5]. On the other hand, the higher ductility of the β titanium seemed to be a cause for its poor wear resistance. In this investigation [5], the authors introduced copper to Ti6Al4V to produce an alloy with an increased resistance to plastic deformation and thus improved the wear resistance of the new alloy.

Extensive research has been performed in ambient conditions to study the frictional performance of conventional titanium alloys Ti6Al4V along with other types of titanium alloys under different test conditions [3,9,20–22]. In dry air, friction and wear behaviour of titanium alloys Ti6Al4V was evaluated using reciprocating-sliding pin on flat configuration [3]. It was found that surface deformation behavior and transfer characteristics can control the friction behavior of titanium alloys. In more recent work [22], the effect of varying the atmospheric gas (N_2 and CO_2) on the friction behavior of aluminum and titanium were studied and it was found that N_2 and air behaved statistically identical for both metals whereas CO_2 increased friction for aluminum but decreased it for titanium. Other researches have established that in ambient conditions severe adhesive wear occurs between metals and the friction coefficients can exceed unity [23].

Recently, it was shown that sliding Ti6Al4V against GCr15 steel counterface can rise the interface temperature up to 800 °C [20]. Besides, an increase in the PV factor can increase both interface temperature and wear rate and decrease the friction coefficient. It was also reported that such conditions ($0.42T_m$, melting temperature) result in damages of different features on the worn surfaces of Ti6Al4V, i.e. severe plastic deformation, thermal softening and melting damages. Therefore controlling the temperature at the interface by advancing the heat-transfer capability [20] or introducing cryogenic temperature at the interface would control the friction coefficient. This is one of the main objectives of the current work. Cryogenic fluids are used nowadays to replace conventional cutting fluids [24,25] due to environmental contaminants and health hazards reasons. Two types of cryogen are commonly used in machining, namely liquid carbon dioxide (LCO_2) and liquid nitrogen (LN_2). The boiling points for both gases are −78.5 °C and −196 °C. Both gases are abundant and can be recycled; they can be compressed to liquid form, cool the cutting tools and evaporate and become gas again. However, at ambient temperature, carbon dioxide is heavier than air; therefore carbon dioxide can accumulate at the floor of the workshop and cause threats to the workers. As a result, carbon dioxide is not encouraged to use as a cryogen in machine shop. On the other hand, nitrogen (LN_2) offers multiple benefits as it provides higher cooling effect compared to carbon dioxide (boiling point of N_2 is lower than CO_2). Therefore, LN_2 can remove heat effectively from the cutting zone and hence lower the cutting temperatures [26]. Moreover, LN_2 can modify frictional characteristics at the tool/chip interface and changes the material properties of workpiece and tool. In addition nitrogen is abundant (79% of air), it is therefore affordable to use. It can be compressed to a liquid and as a liquid cools the tool, evaporates and becomes part of the air again. Thus it is naturally recycled without damage to the environment. For these reasons LN_2 is preferred as a cryogenic fluid.

From the above review, it can be realized that previous work has focused on various approaches to improve friction and wear properties, none of which is directly comparable to the current work. These approaches include heat treatments, surface coating and modification, introduction of new composition of Ti-alloy, controlling the ambient condition (in dry air, N_2, CO_2), and one of the most recent approaches was controlling the capability of

Table 1
Chemical composition of Ti64 and Ti54 alloys [27,28].

Alloy	Al	V	Mo	Fe	C	O	N	Ti
Ti–6Al–4 V	5.5–6.75	3.5–4.5	–	0.40 max	0.08 max	0.20 max	0.05 max	Balance
Ti–5Al–4 V–0.6Mo–0.4Fe	4.5–5.5	3.0–5.0	0.4–1.0	0.2–0.8	0.1 max	0.2 max	0.03 max	Balance

Table 2
Properties of titanium alloys [27,28].

Titanium alloys	Ti64	Ti54
Phase	$\alpha+\beta$	$\alpha+\beta$
Density (g/cm^3)	4.42	4.44
Ultimate tensile strength (MPa)	965	NA
Thermal conductivity (W/m K)	6.6 @ 20 °C	6.96 @ 25 °C
Specific heat (J/g K)	0.580 @ °C	0.54 @ 25 °C
Young's modulus (GPa)	107–122	NA
Shear modulus (GPa)	41–45	NA

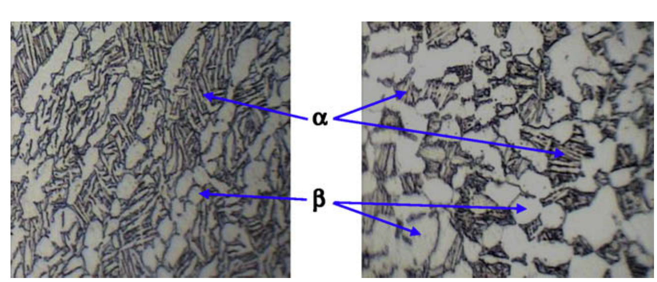


Fig. 1. (a) Microstructure of Ti54 and (b) microstructure of Ti64.

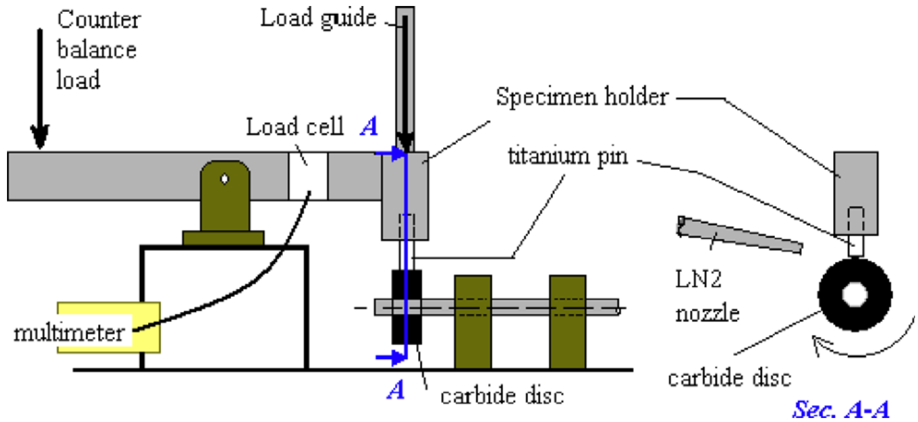


Fig. 2. Schematic diagram of cryogenic pin-on-disc apparatus.

Table 3
Surface roughness and hardness of titanium alloys and carbide wheel.

Materials	Ra before test (μm)	Ra after test (μm)	Hardness
Ti–6Al–4 V pin	0.095–0.361	0.724–1.089	350 HV10 (equivalent to 69 HRA)
Ti–5Al–4 V–0.6Mo–0.4Fe pin	0.083–0.148	0.466–0.669	325 HV10 (equivalent to 68 HRA)
Tungsten carbide wheel	1.157–1.169	0.459–0.536	92 HRA

heat transfer of Ti-alloy. However the closest approaches to the current work are the introduction of a new type of Ti-alloy and controlling the ambient conditions. As it appears, none of the past researches gave attention to investigate the frictional behaviour of Ti-alloy sliding against tungsten carbide under cryogenic conditions. Moreover, no information was available about the effect of test parameters' interactions (i.e. the combined effects of load/speed, load/sliding time, speed/sliding time) on the frictional behaviour of Ti alloy. All published work focused only on the study of the effect of one-parameter-at a time. Thus the current work aims to achieve two main objectives. The first is to evaluate the friction coefficient of newly developed titanium alloy Ti–5Al–4V–0.6Mo–0.4Fe (Ti54) (supplied by Timet Inc., USA) sliding against tungsten carbide wheel and compare with the conventional titanium alloy Ti6Al4V (Ti64). The second objective is to study the effect of cryogenic conditions on the frictional behaviour of both titanium alloys along with the effect of main and interaction of test parameters. The main parameters are load, speed, and test duration. To reduce the experiment activities and maximize result quality, a statistical approach based on design of experiment is implemented using a factorial design of 2^3. Response surface methodology (RSM) is used to develop predictive models for frictional behaviour of the titanium alloys at both room and cryogenic temperatures.

2. Experimental work

2.1. Tested materials

In this research, two types of titanium alloys were tested at room and cryogenic environments; conventional Ti6Al4V alloy and newly developed Ti–5Al–4V–0.6Mo–0.4Fe alloy, supplied by Titanium Metals Inc. (TIMET), USA. These alloys will subsequently be referred to as Ti64 and Ti54. Details of the chemical composition and material properties are given in Tables 1 and 2 [27,28]. Fig. 1 shows the microstructures of the α- and β-phase on the cross sections of Titanium alloys Ti54 and Ti64, respectively.

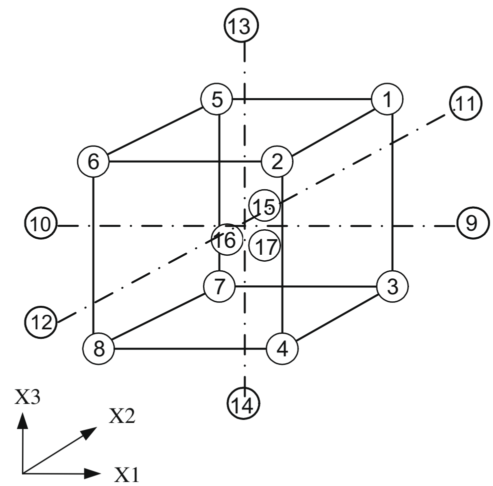


Fig. 3. Central composite design (CCD).

Table 4
Levels of independent variable for experiment.

Level	Lowest	Low	Center	High	Highest
Coding	−1.682	−1	0	1	1.682
x_1, speed (m/s)	0.1295	0.3	0.55	0.8	0.9705
x_2, load (N)	6.464	9.81	14.715	19.62	22.956
x_3, time (min)	2.636	4	6	8	9.364

2.2. Experimental set-up and tribo test procedure

Tribo tests were conducted using a pin-on-ring configuration, shown in Fig. 2, at two different environment conditions, i.e. at room (dry) and cryogenic (liquid nitrogen, LN_2) conditions. Tribo specimens of titanium alloys were machined from plates of size 147 mm × 155 mm × 16 mm into cuboid pins of size 5 mm × 5 mm × 20 mm. At the beginning, rubbing faces of all pins (5 × 5 mm^2) were polished to initial surface roughness ranging between 0.08-to-0.361 μm Ra, Table 3. During tests, the rubbing faces of the titanium alloy pins were rubbed against a tungsten carbide wheel (counterface) having 25 mm diameter, 9 mm thickness, and hardness of 92 HRA (detailed specifications of the carbide disc are

Table 5
Matrix of experiments with average friction coefficients for central composite design.

	Input			Response			
	Speed, S	Load, L	Time, T	Average COF			
				Ti64D[a]	Ti64C[a]	Ti54D[a]	Ti54C[a]
1	1	1	1	0.6149	0.5814	0.8449	0.5898
2	1	−1	1	0.5432	0.4513	0.4847	0.5098
3	1	1	−1	0.5948	0.4367	0.5647	0.5797
4	1	−1	−1	0.5265	0.4062	0.5566	0.4663
5	−1	1	1	0.6023	0.4852	0.5270	0.4727
6	−1	−1	1	0.4764	0.2925	0.4764	0.2507
7	−1	1	−1	0.5948	0.3840	0.5421	0.3990
8	−1	−1	−1	0.4513	0.4513	0.4964	0.3159
9	1.682	0	0	0.5587	0.5945	0.5731	0.5229
10	−1.682	0	0	0.5516	0.4298	0.5158	0.4871
11	0	1.682	0	0.5556	0.5923	0.5464	0.6061
12	0	−1.682	0	0.4566	0.4566	0.4566	0.4566
13	0	0	1.682	0.5731	0.5444	0.6160	0.3486
14	0	0	−1.682	0.5014	0.5014	0.5892	0.4764
15	0	0	0	0.5659	0.5945	0.5802	0.4155
16	0	0	0	0.5945	0.4155	0.5731	0.4513
17	0	0	0	0.5516	0.4943	0.6017	0.5014

Referring to sliding speed $\times 1 \Rightarrow S$, Normal Load $\times 2 \Rightarrow L$, and Sliding time $\times 3 \Rightarrow T$.
[a] D for dry and C for Cryogenic. The response y denotes to the coefficient of friction COF.

Table 6
ANOVA for dry friction of Ti64D (full model).

Source	DF	Seq SS	Adj SS	Adj MS	F	P	
Regression	9	0.035253	0.035253	0.003917	5.11	0.021	(Significant)
Linear	3	0.028963	0.007591	0.002530	3.30	0.087	
Square	3	0.004165	0.004165	0.001388	1.81	0.233	
Interaction	3	0.002124	0.002124	0.000708	0.92	0.477	
Residual error	7	0.005361	0.005361	0.000766			
Lack of fit	5	0.004403	0.004403	0.000881	1.84	0.389	(Non-significant)
Pure error	2	0.000958	0.000958	0.000479			
Total	16	0.040613					

Table 7
Estimated regression coefficients for dry friction of Ti64D (full model).

Term	Coef	SE Coef	t	P	
Constant	0.017878	0.181675	0.098	0.924	
Speed, S	0.255303	0.222503	1.147	0.289	
Load, L	0.039446	0.012611	3.128	0.017	
Time, T	0.031180	0.030929	1.008	0.347	
S^2	−0.016809	0.131871	−0.127	0.902	(Non-significant)
L^2	−0.000765	0.000343	−2.232	0.061	
T^2	−0.001846	0.002060	−0.896	0.400	
$S*L$	−0.013203	0.007979	−1.655	0.142	
$S*T$	0.001048	0.019568	0.054	0.959	(Non-significant)
$L*T$	−0.000181	0.000997	−0.181	0.861	(Non-significant)

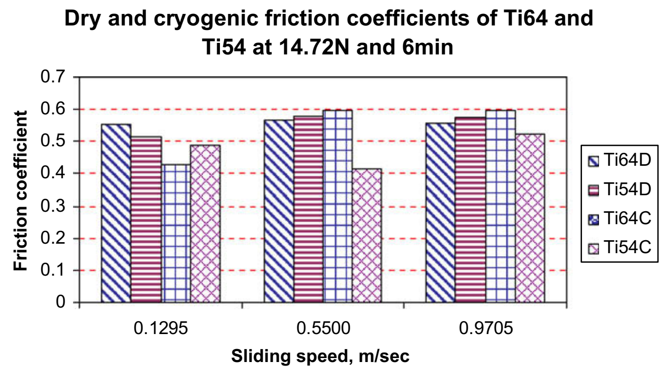


Fig. 4. Effect of sliding speed on dry and cryogenic COF of Ti64 and Ti54 alloys.

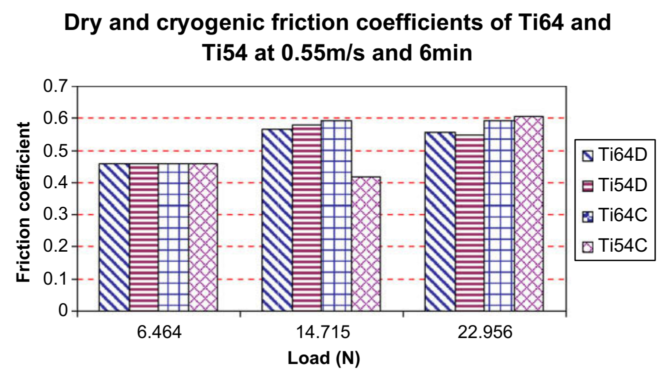


Fig. 5. Effect of load on dry and cryogenic COF of Ti64 and Ti54 alloys.

given elsewhere [29]. The surface roughness of the friction track on the carbide wheel was also measured before tests and found to be in the range of 1.157–1.169 μm Ra. Surface roughness measurements for both titanium specimens and tungsten carbide were performed before and after tests using Mahr Perthometer S2 Profilometer and reported in Table 3. The hardnesses of the titanium pins and carbide wheel are also given in Table 3. The rotating speed of the counterface can be adjusted through a variable speed DC motor control (KB Penta Power KBMD-240D). The normal load was applied by using dead weight through load guide (shown in Fig. 2). In this study three independent variables with five levels were selected, i.e. normal loads (6.4, 9.81, 14.72, 19.62, 22.96 N), sliding speed (0.1295, 0.3, 0.55, 0.8, 0.971 m/s), and sliding time (2.64, 4, 6, 8, 9.36 min). Frictional force at the sliding interface of the specimen was monitored and recorded every 15 s for the first minute and every 30 s for the rest of the test using a load cell (ACCUTEC load cell L6J) attached to the load lever. The average coefficient of friction (COF) was calculated by dividing the average frictional force for each test by the applied normal load. Two sets of

Table 8
ANOVA for dry friction, Ti64D (reduced model).

Source	DF	Seq SS	Adj SS	Adj MS	F	P	
Regression	6	0.035213	0.035213	0.005869	10.87	0.001	(Significant)
Linear	3	0.028963	0.010194	0.003398	6.29	0.011	
Square	2	0.004153	0.004153	0.002076	3.85	0.058	
Interaction	1	0.002097	0.002097	0.002097	3.88	0.077	
Residual error	10	0.005400	0.005400	0.000540			
Lack of fit	8	0.004442	0.004442	0.000555	1.16	0.542	(Non-significant)
Pure error	2	0.000958	0.000958	0.000479			
Total	16	0.040613					

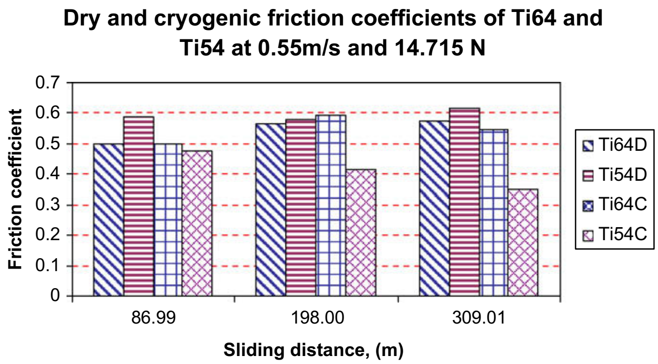


Fig. 6. Effect of sliding distance on dry and cryogenic COF of Ti64 and Ti54 alloys.

experiments were performed; the first was conducted in the air at room temperature under dry condition while the second, was conducted in the liquid nitrogen (LN_2). The LN_2 jet was directed to the interface between the titanium alloy pin and the counterface disc using a nozzle. To eliminate errors in the measured friction forces caused by the liquid nitrogen jet, the liquid nitrogen was injected to the interface before starting the test and the frictional force reading was reset to zero. The results of COF are presented and discussed in the present study whereas the wear results are discussed in our next work.

3. Response surface methodology (RSM)

Response surface methodology (RSM) is practical, economical and relatively easy to use. RSM approach is a combination of statistical and mathematical techniques useful for developing, improving and optimizing processes [27]. This method has been used by some other researchers for prediction of tool life, surface roughness, wear resistance, etc. [30]. It is commonly applied in situations where several input variables potentially influence the response (output) of interest.

A polynomial model of second order type was proposed to represent the relationship between friction coefficient and tribo test independent variables. The performance of the model depends on a large number of factors that can act and interact in a complex manner. In the present work, the input variables are sliding speed (x_1), load (x_2) and sliding time (x_3) and the output (response) is friction coefficient (y). A response surface model is usually expressed as:

$$y = \beta_0 + \sum_{i-1}^{k} \beta_i x_i + \sum_{i=1}^{k} \beta_{ii} x_i^2 + \sum_{i=1}^{k-1} \sum_{j=1}^{k} \beta_{ij} x_i x_j + \varepsilon \qquad \text{for} \quad i < j \qquad (1)$$

where β_o, β_i ($i = 1, 2, \ldots, k$) and β_{ij} ($i = 1, 2, \ldots, k$, $j = 1, 2, \ldots, k$) are the unknown as regression coefficients to be estimated by using the method of least squares. In this equations ε are experimentally random errors and $x_1, x_2, \ldots, x_k$ are the input variables that influence the response y, k is the number of input factors. A second order model with two-factor interaction (2FI) is chosen so that the

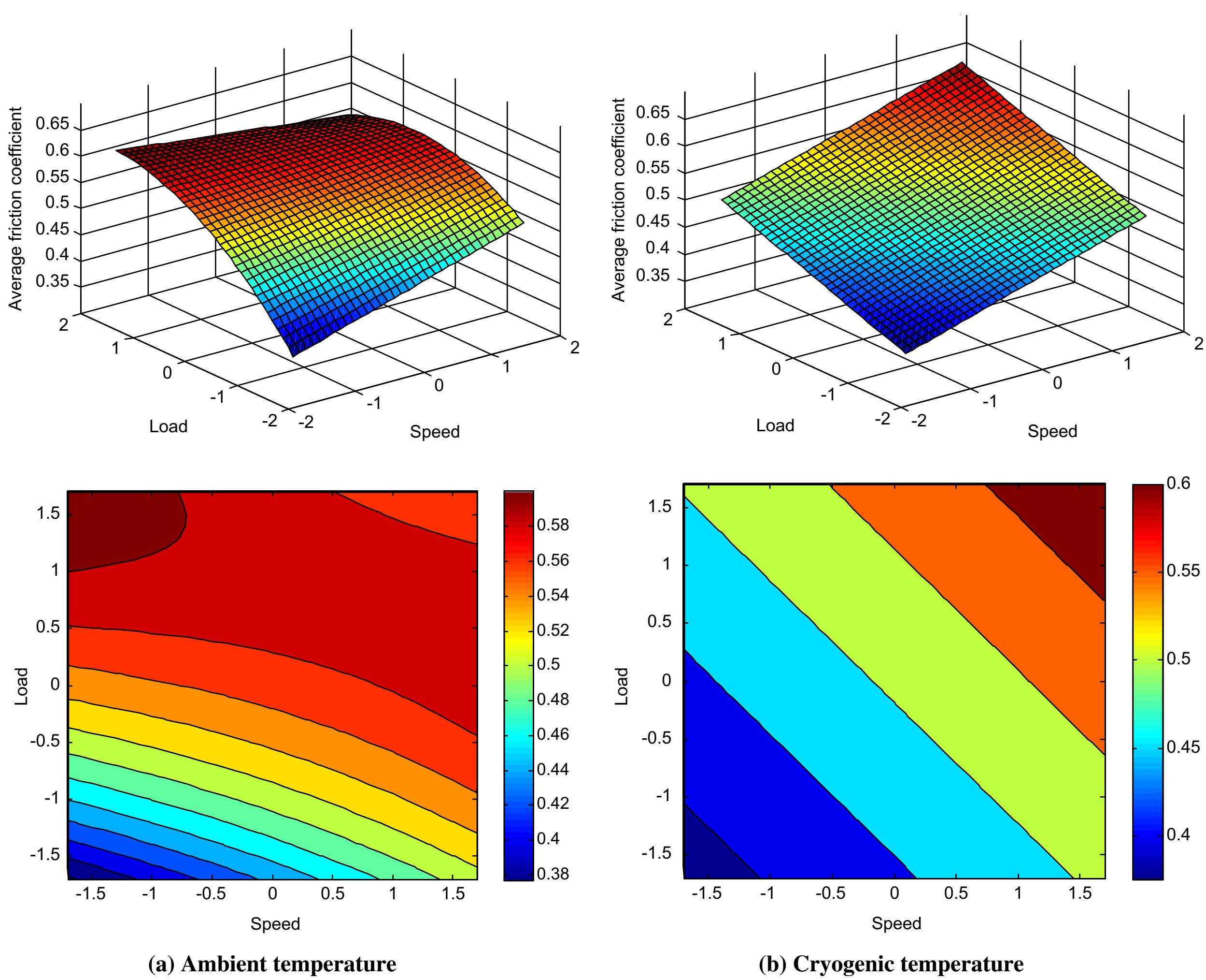


Fig. 7. Response surface and contour plots: effect of load and speed on average COF of Ti64D and Ti64C.

two-factor interaction (2FI) can be analyzed. It should be realized that if higher order model (e.g. cubic) were chosen then it would be able to analyze three-factor interactions (3FI) instead of 2FI. However, from the literature [31] it was shown that second order model led to a reasonable approximation and was able to predict COF and wear rate of PCTFE/A12024 under LN_2 as a function of pressure and sliding speed. In the current work, the matrix approach was adopted to solve Eq. (1) and the regression coefficients are calculated using MATLAB software.

3.1. Design of experiments (DoEs)

The concept of using design of experiment is to reduce the test activity and maximize the result quality. Compared to conventional one-factor-at-a-time technique, factorial design is considered more efficient. It enables the study of both main and interaction effects among the different variables. A design of experiment consisting of 17 experiments is used to develop predictive equations for friction coefficient. The design consists of a factorial portion 2^k, a center point and an axial portion (star points). The factorial portion is eight experiments represent a 2^3 factorial design with factor levels coded by −1 and +1 (located at the vertices of Central Composite Design, CCD) as shown in Fig. 3. Three experiments represent an added center point to the CCD, repeated three times to estimate the pure error. The axial portion (star points) is a point located on the coordinate axes of the factorial portion at an augment distance $\alpha = 1.682$ from the design center. The augment point (distance) was chosen depending on the capacity of tribo test machine, which has three levels for each of the independent variables denoted by −1.682, 0, +1.682. The coded and natural levels of the independent variables are presented in Table 4. As mentioned earlier, the experiment includes three independent variables whose five levels are coded according to the transforming Eq. (2) for each of the independent variables:

$$x_1 = \frac{speed - 0.55}{0.25}; \quad x_2 = \frac{load - 14.715}{4.095}; \quad x_3 = \frac{time - 6}{2} \tag{2}$$

Table 5 shows the design matrix and the experimental results for both alloys in dry (D) and cryogenics (C) conditions.

3.2. Development of friction coefficient models

By using the factorial design, a total of 17 experiments were conducted and regression coefficients were calculated. The full models for average friction coefficients of Ti64 and Ti54 for each sliding condition, i.e. dry and cryogenic, were obtained as:

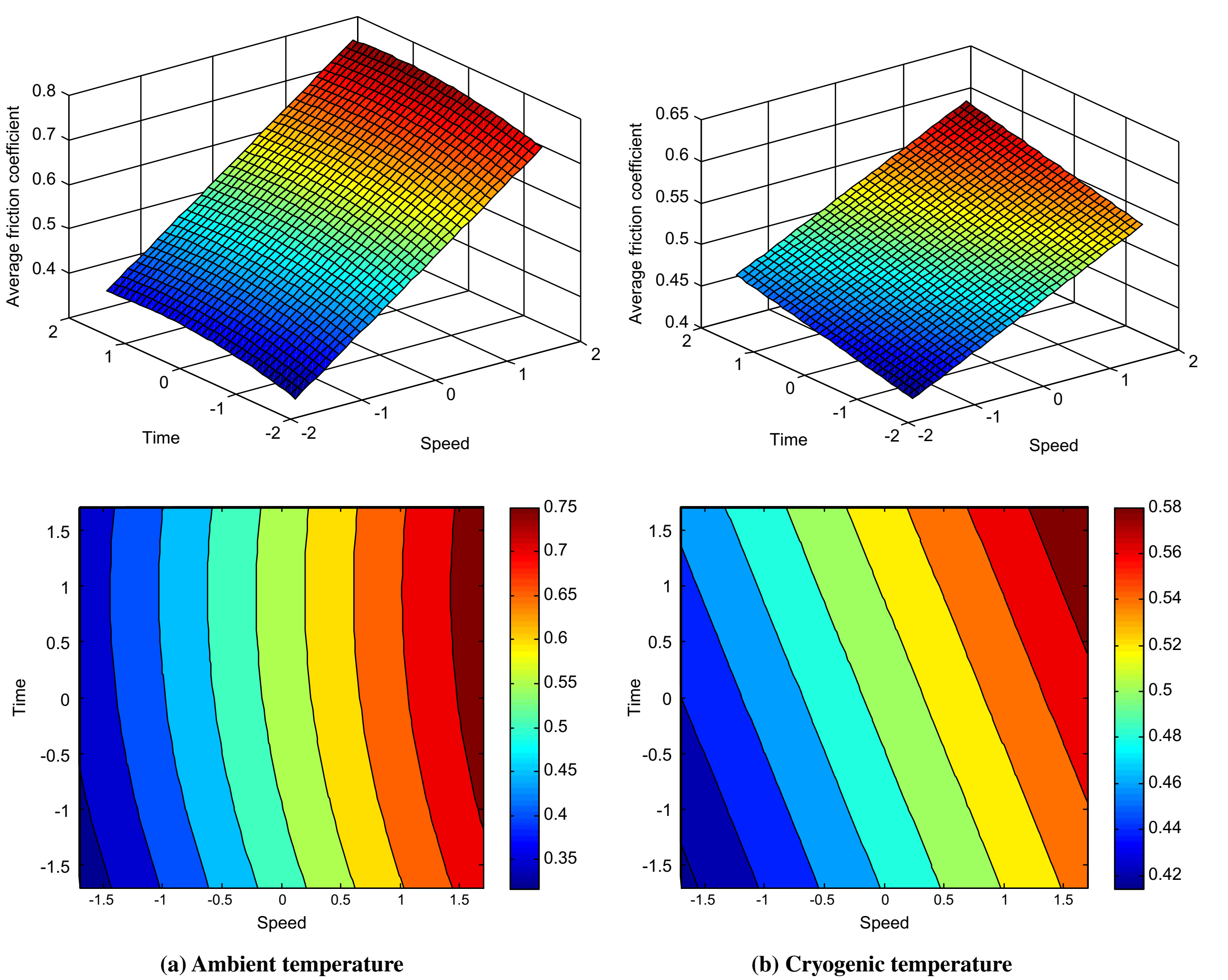


Fig. 8. Response surface and contour plots: effect of speed and time on average COF of Ti64D and Ti64C.

- *For Ti64 alloy under dry and cryogenic sliding conditions are given as:*

$$\mathrm{COF}_{D_{64}} = 0.5694 + 0.0122S + 0.0422L + 0.0139T - 0.0011S^2 - 0.0184L^2 - 0.0074T^2 - 0.0162SL + 0.0005ST + 0.0018LT \quad (3)$$

$$\mathrm{COF}_{C_{64}} = 0.5069 + 0.0395S + 0.0377L + 0.015T - 0.015S^2 - 0.0107L^2 - 0.0122T^2 + 0.0044SL + 0.0309ST + 0.045LT \quad (4)$$

- *For Ti54 alloy under dry and cryogenic sliding conditions are given as:*

$$\mathrm{COF}_{D_{54}} = 0.5841 + 0.037S + 0.0451L + 0.016T - 0.0112S^2 - 0.0264L^2 + 0.0094T^2 + 0.034SL + 0.0304ST + 0.0446LT \quad (5)$$

$$\mathrm{COF}_{C_{54}} = 0.4581 + 0.0582S + 0.0549L - 0.0112T + 0.0092S^2 - 0.0185L^2 - 0.0235T^2 - 0.014SL + 0.0056ST + 0.0132LT \quad (6)$$

3.3. Analysis of variance (ANOVA)

Analysis of variance (ANOVA) was performed to determine the significant and non-significant parameters as well as the validity of the full models, Eqs. (3)–(6). The ANOVA was carried out on each model for a confidence level of 95%. The results of ANOVA performed on the full model of average friction coefficient of Ti64 ($\mathrm{COF}_{D_{64}}$) at dry condition (Eq. (3)) are listed in Tables 6 and 7.

In Table 6, the value of 'P' is less than 0.05. This is desirable as it indicates that the terms in the model have a significant effect on the response [32]. The ANOVA (Table 6), demonstrates that the model is highly significant, and the lack of fit is non-significant. Furthermore, the significance of each coefficient in the full model was examined by the t-values and P-values and the results are listed in Table 7. According to [33], larger values of t-test and smaller values of "P" indicate that the corresponding coefficient is highly significant. Hence, the results given in Table 7 suggest that the influence of Speed × Speed (S^2), Speed × Time ($S * T$) and load × time ($L * T$) are non-significant and therefore can be removed from the full model to further improve the model. By doing so, the full model for the ($\mathrm{COF}_{D_{64}}$) can be reduced as:

- *For dry friction coefficient of Ti64D*

$$\mathrm{COF}_{D_{64}} = 0.5694 + 0.0122S + 0.0422L + 0.0139T - 0.0184L^2 - 0.0074T^2 - 0.0162SL \quad (7)$$

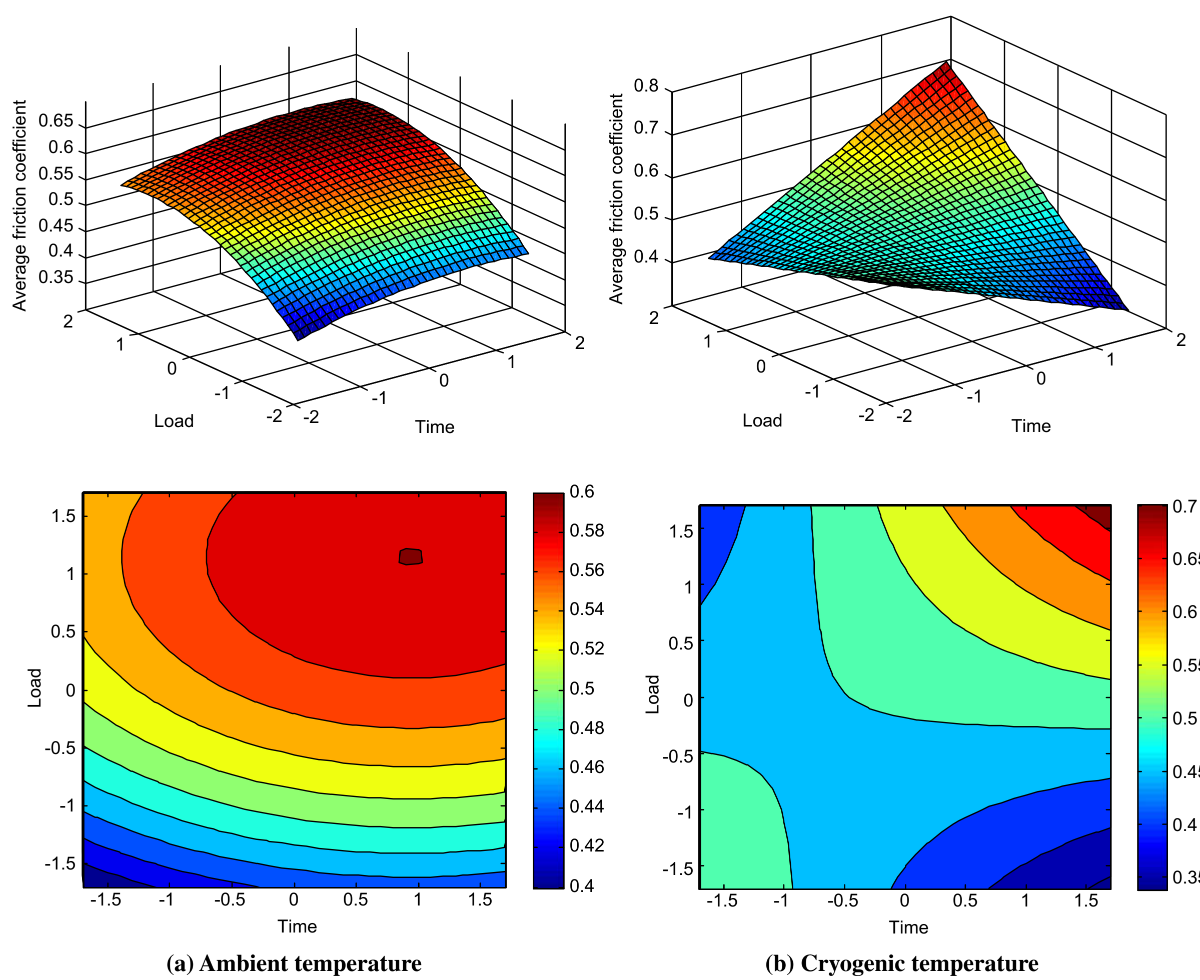


Fig. 9. Response surface and contour plots: effect of load and time on average COF of Ti64D and Ti64C.

ANOVA was performed on the reduced model and the results are presented in Table 8. From that Table 8, it can be seen that, the reduced model is improved as the *P*-value for regression is only 0.001 compared to 0.021 for the full quadratic model (Table 6). Thus, Eq. (7) represents the coded form of final empirical model for dry friction coefficient of Ti64.

The above analyses were repeated for each of the full model given in Eqs. (4)–(6), i.e. for Ti64C, Ti54D and Ti54C and the final reduced models were obtained as:

- *For cryogenic friction coefficient of Ti64C*

$$COF_{C_{64}} = 0.5069 + 0.0395S + 0.0377L + 0.015T + 0.045LT \quad (8)$$

- *For dry friction coefficient of Ti54D*

$$COF_{D_{54}} = 0.5841 + 0.037S + 0.0451L + 0.016T - 0.0264L^2 + 0.034SL + 0.0446LT \quad (9)$$

- *For cryogenic friction coefficient of Ti54C*

$$COF_{C_{54}} = 0.4581 + 0.0582S + 0.0549L - 0.0112T - 0.0185L^2 - 0.0235T^2 \quad (10)$$

It should be noted that the above equations are valid over the range of tested conditions 0.1295 < speed < 0.9705 m/s; 6.464 < load < 22.956 N; 2.636 < sliding time < 9.364 s for sliding the titanium alloys (Ti64D, Ti64C, Ti54D, and Ti54C) on counterface of tungsten carbide.

4. Results and discussion

Figs. 4–6 compare the effects of main parameters (speed, load, and sliding distance) on the dry and cryogenic measured friction coefficients of Ti64 and Ti54 alloys sliding against tungsten carbide wheel. Whereas the effects of the parameter interactions (load/speed, time/speed, and load/time) in the form of response surfaces and contour plots (projection of the constant-value lines of the response surface on *x*–*y* plane) on the average COF of Ti64 and Ti54 are shown in Figs. 7–12. The results in Figs. 7–12 were produced by using Eqs. (7)–(10). As shown in Table 5 the experimental values of friction coefficient of Ti64D and Ti64C are in the range of (0.451–0.615) for dry and (0.293–0.58) for cryogenic conditions. Depending on test condition, the introduction of LN_2, dropped the range of COF by 35–5%. Previous work [22] similarly showed that the introduction of CO_2 decreased the COF of Ti6Al4 V from 0.41 to 0.33 (19%) and introduction of dry N_2 decreased the COF from 0.41 to 0.39 (5%). The lower value of dry COF is comparable to the published value (0.41) [22].

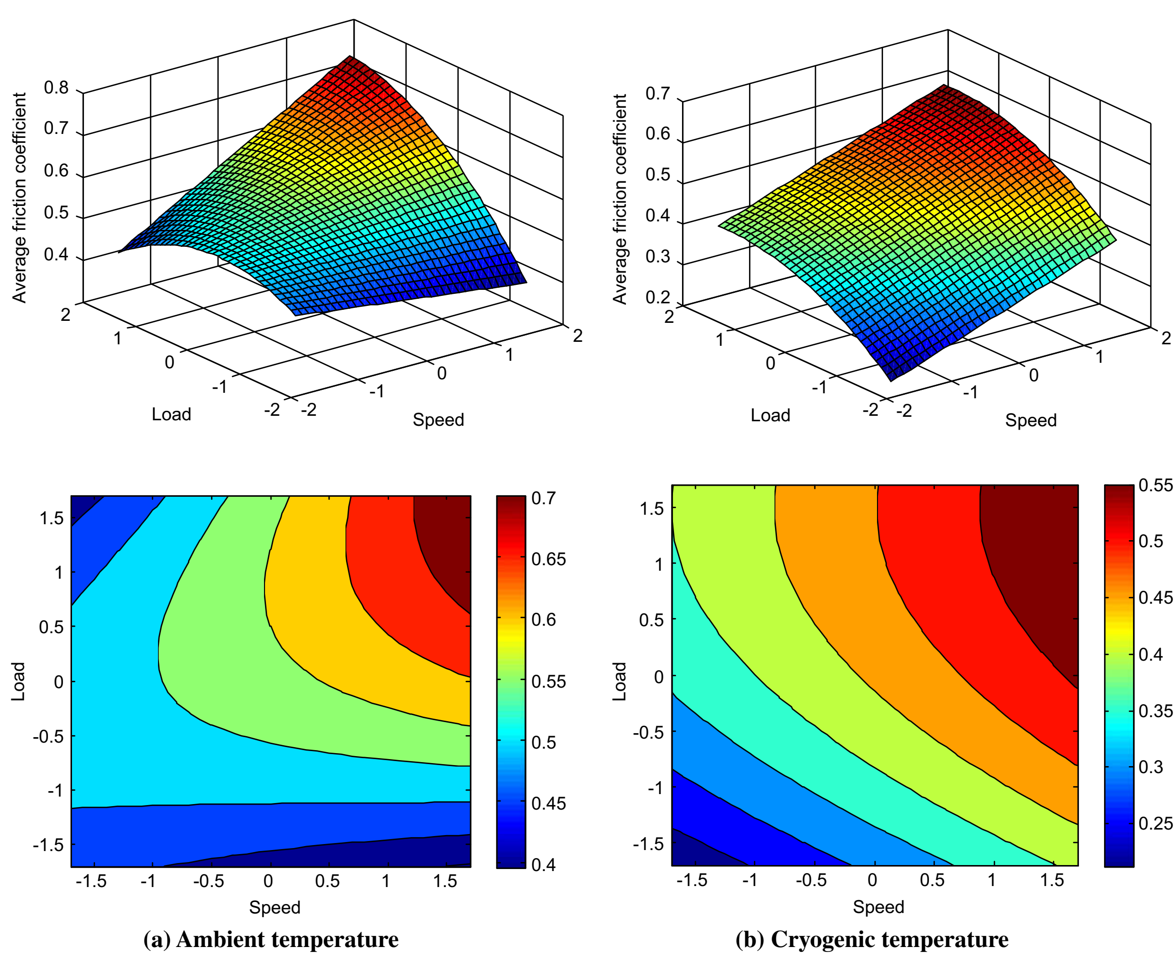


Fig. 10. Response surface and contour plots: effect of load and speed on average COF of Ti54D and Ti54C.

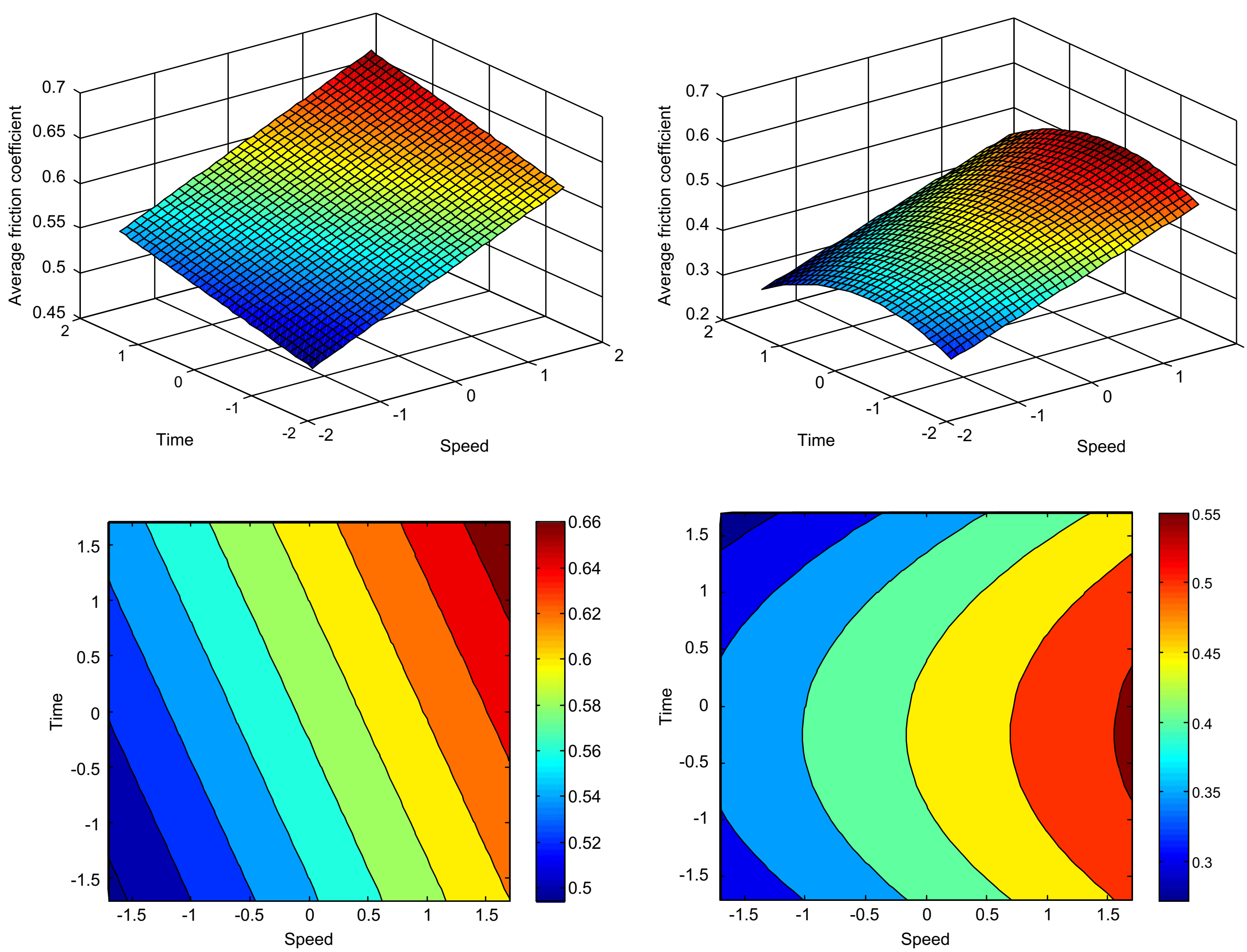
4.1. Effect of the main parameters on dry and cryogenic COF of Ti64 and Ti54

There is a general recognition that friction and wear characteristics are strongly dependent on load, sliding speed, sliding time (distance), temperature, contact geometry, surface roughness, ambient atmosphere and material surface compositions. To the best of the authors' knowledge, no information were available in the literature about COF of conventional Ti6Al4V/tungsten carbide pairs and the effect of cryogenic condition on the COF of Ti6Al4V alloy sliding against tungsten carbide. That means none of the published work is directly comparable to the current work. However, in this following section, an attempt is made to present such information and highlight on some previously published results for the conventional Ti64 tested against other counterface under dry conditions.

As shown in Figs. 4–6, sliding the Ti alloys against the tungsten carbide wheel in air produced COF in the range of 0.45–0.58 for Ti64 and 0.45–0.6 for Ti54 (Figs. 4–6). For Ti64 under dry sliding, slight increase of COF was observed with increasing load or sliding distance (time), Figs. 5 and 6 and no dependence on speed was observed (Fig. 4). The absence of the speed effect on the friction of Ti6Al4V may be related to the decreased flow stress sensitivity of this material to applied strain rate [3].

The effect of cryogenic conditions on COF of both Ti64 and Ti54 (Figs. 4–6) was expected to reduce the COF significantly compared to those obtained at ambient temperature. According to the published work [18], sliding at ambient conditions generates a large quantity of frictional heat at the contact surface and the interface temperature will rise up to a higher value, depending on the combined effect of speed and load, even up to melting temperature of materials [20]. This is true for titanium alloys because of poor heat-transfer capability. Therefore, in the current work, it was thought that the introduction of LN_2 to the Ti/tungsten carbide interface would reduce COF. This was the case for Ti54 but not for Ti64. Previous study on Ti6Al4V [20] indicated that there is a critical temperature for Ti6Al4V alloys at which friction coefficient decreases. However, observing the results Figs. 4–6, one can see that there was a slight increase of the COF of Ti64 only at higher levels of sliding speed and load. In contrary, the COF of Ti54 shows a substantial decrease upon the introduction of LN_2.

The observed increasing trend of dry and cryogenic COF of Ti64 and dry COF of Ti54 with load and sliding velocity (Figs. 4 and 5) is in agreement with earlier published work [20] for COF of Ti6Al4V/GCr15 pairs.



(a) Ambient temperature (b) Cryogenic temperature

Fig. 11. Response surface and contour plots: effect of speed and time on average COF of Ti54D and Ti54C.

4.2. Effect of the interaction parameters on dry and cryogenic COF of Ti64 and Ti54

4.2.1. Surface plots and contour of titanium alloy Ti64

Results of the combined effects of load and speed on the COF of Ti64D at room and cryogenic temperatures are presented in Fig. 7. For dry condition, Fig. 7 shows clearly that the predictive COF of Ti64D increases significantly with speed and load and the highest value occurs at lowest speed and highest load. It can be also observed that the COF behaves linearly with speed and nonlinearly with load.

In cryogenic condition, combination of load and speed still play an important role in influencing average COF of Ti64C (Fig. 7b). The highest value of COF of Ti64C occurs at highest values of load and speed. In contrary to dry sliding, the COF, in cryogenic condition, behaves linearly with both load and speed.

The influence of speed and time on the predicted COF of Ti64D and Ti6dC is presented in Fig. 8. Both surface and contour plots show clearly that a substantial linear increase of the COF with speed and a slight nonlinear increase with the sliding time. In cryogenic condition, the increase in speed still resulted in increased COF (Fig. 8b). This trend is similar to dry condition. However, the influence of sliding time on COF in cryogenic become more significant compared to dry condition. This seems to be due to the cooling effect of liquid nitrogen which is to harden the sliding surfaces of titanium alloys and tungsten carbide [26,34], and this effect increases with time as the materials become cooler. This in turn causes the increase of COF with time.

Fig. 9 confirms the nonlinearity behaviour of COF with load for Ti64D observed in Fig. 7. The figure also indicates the significant effect of increasing the load on COF of Ti64D. From the contour plot (Fig. 9a), it can be observed that the highest COF of Ti64D is predicted at load ≈ 1 (19.62 N) and time ≈ 1 (8 min). Fig. 9b shows the relationship between load, speed and average COF of Ti64C on which the highest COF occurs at the highest load and highest sliding time whereas the lowest COF occurs at highest time and lowest load.

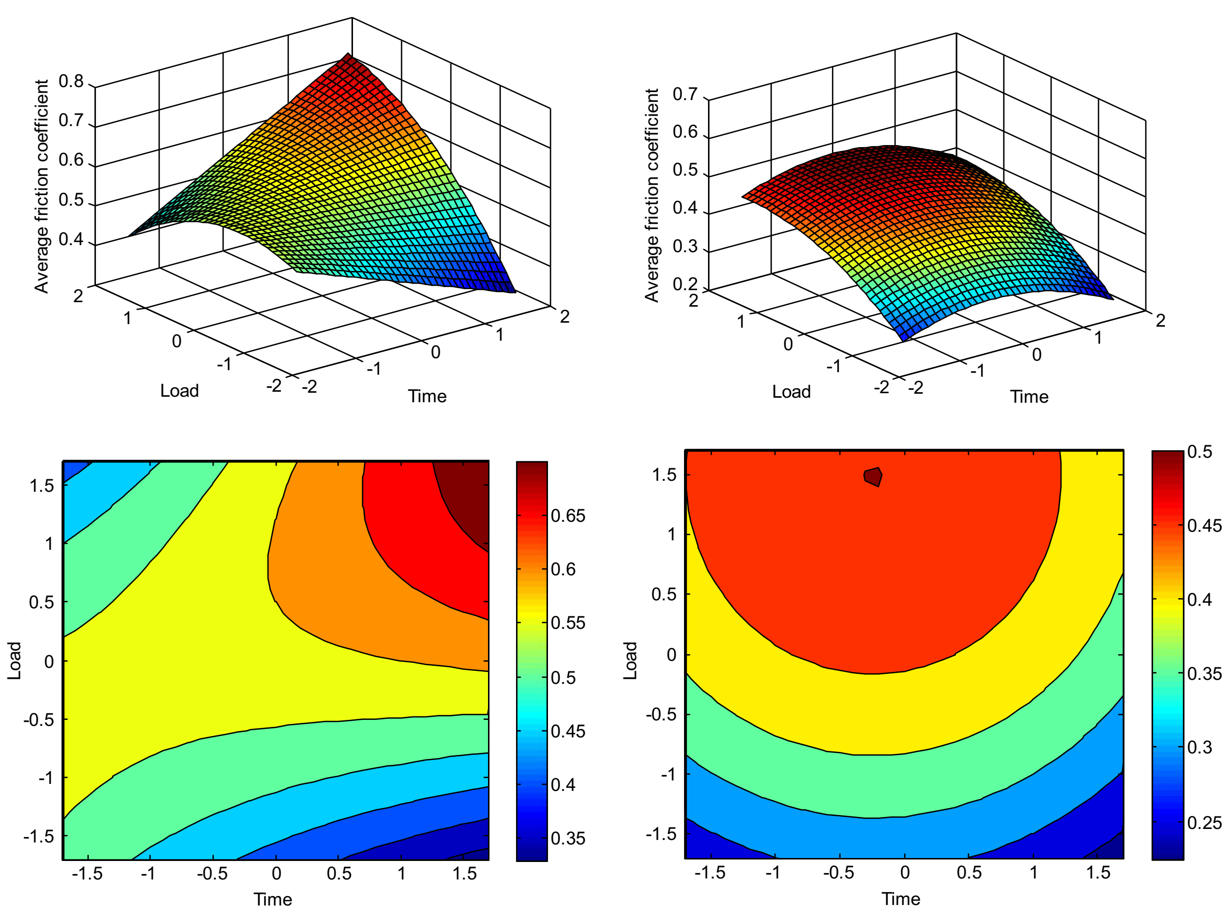
4.2.2. Surface plots and contour of titanium alloy Ti54

For modified titanium alloy Ti54, the effects of speed, load and time on COF at ambient and cryogenic temperatures are shown in Figs. 10–12. Cryogenic environment resulted in a decrease in the highest values of COF (Figs. 10b, 11b, and 12b) compared to those predicted at room temperature (Figs. 10a, 11a, and 12a).

For dry condition, COF of Ti54D increases with increasing speed at load higher than −0.5 (12.67 N) and the highest COF occurs at the highest values of both speed and load, Fig. 10a. Under cryogenic conditions (Fig. 10b), the COF increases nonlinearly with the load and linearly with the speed but with lesser extent compared to the dry COF.

Fig. 11 shows that for both dry and cryogenic conditions, speed is more influential as compared to time, i.e. increasing speed substantially increases the COF whereas the test duration has less effect on increasing the COF.

The combined effect of speed–time on average COF Ti54C is presented in Fig. 11b. At low speed, the average COF is low. An



(a) Ambient temperature **(b) Cryogenic temperature**

Fig. 12. Response surface and contour plots: effect of load and time on average COF of Ti54D and Ti54C.

Table 9
Comparisons between predicted upper values of COF of titanium alloys Ti64 and Ti54 at dry and cryogenic conditions.

Variables	At room temperature Ti64	At cryogenic temperature Ti64	At room temperature Ti54	At cryogenic temperature Ti54	Remarks
Load/speed	0.6 At highest load and lowest speed	0.62 At highest load and lowest speed	0.71 At highest load and speed	0.58 At highest load and speed	At cryogenic temperature, COF increased by 3% for Ti64 and decreased by 18% for Ti54
Time/speed	0.75 At highest time and speed	0.58 At highest speed and time	0.67 At highest speed and time	0.51 At highest speed and lowest time	At cryogenic temperature, COF decrease by about 22% for Ti64 and 23% for Ti54
Load/time	0.58 At highest load and time	0.7 At highest load and time	0.7 At highest load and time	0.48 At highest load and lowest time	At cryogenic temperature, COF increased by about 20% for Ti64 and decreased by 31% for Ti54

increase in the sliding speed results in an increase in average COF which is unusual finding since most of the literature reports opposite trend [35,36]. The effect of sliding speed on average COF is not that significant

The combined effect of load–time on average COF of Ti54D is shown in Fig. 12a. It is clear that COF increases with an increase in time for high load region (above than 0.5) and increases with an increase in load for long sliding time (above than 0.5). The highest average friction occurred when both load and time are high. Fig. 12b shows that COF of Ti54C increases with load and this increasing tendency remains even at higher loads. Beside this, COF of Ti54C did not influence much by increasing the sliding distance (sliding time) for constant load and speed conditions.

Table 9 shows the highest values of COF for both titanium alloys predicted at room and cryogenic temperatures subjected to different loads, sliding speeds, sliding durations. From this table it can be realized that the most significant decrease of cryogenic COF is noted for Ti54 at highest load and moderate time (31% reduction), i.e. from about 0.7 at room temperature to 0.48 at cryogenic temperature. Also and surprisingly, for titanium Ti64, the combined effect of load/speed and load/time under cryogenic condition is increasing the COF by 3% and 20%, respectively. Meanwhile, the cryogenic COF for Ti54 showed consistent decrease compared to those predicted at room temperature for Ti54 as well as those predicted for Ti64 at both room and cryogenic temperature. Furthermore, the summary of the results outlined in the table suggests that cryogenic condition is essential for decreasing the COF for Ti54 but this does not hold for Ti64, i.e. under certain condition, it increases the COF up to 20%.

5. Conclusions

The main conclusions drawn from this study are listed below:

- Factorial design of experiments has been employed to develop second-order polynomial equations for describing dry and cryogenic friction behaviour of titanium alloys slide against tungsten carbide. The relationships of dry and cryogenic COF of Ti64 and Ti54 with applied load, sliding velocity and sliding time have been successfully obtained by using RSM. These models are valid within the ranges of selected experimental parameters of load, speed, and sliding time.
- The effect of one factor at a time revealed that sliding the Ti64 alloy against the tungsten carbide wheel at higher levels of load and speed in air produced COF slightly lower than sliding in cryogenic condition. Conversely, sliding the Ti54 alloy in air showed higher COF compared to those obtained under cryogenic conditions at all tested parameters. Similar conclusions were drawn from the established equations. The established equations indicated that Ti54 tested in dry air exhibited higher COF compared to Ti64 within the selected experimental conditions but at cryogenic conditions exhibited the lowest COF obtained within the frame of the current work.
- The mixed effects of the variables (interaction) were also noticed. The established equations demonstrated that the interaction of load/speed, time/speed, and load time consistently decrease the COF of Ti54. However this conclusion did not hold for Ti64 whereas the COF increased under cryogenic conditions up 20% when the Ti64 was tested at higher range of load and sliding time.
- Among various parameters, the established equations indicated that interaction of loads and speeds was more effective for both Ti64 and Ti54 and have the most substantial influence on the friction. In addition, COF for both alloys was behaved linearly with the speed but nonlinearly with the load

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